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Reaction of
Perfluoroalkylpolyethers
(PFPE) With 440C Stainless
Steel in Vacuum Under
Sliding Conditions at
Room Temperature

Shigeyuki Mori
and Wilfredo Morales
*Lewis Research Center
Cleveland, Ohio*



National Aeronautics
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Summary

Reactions of the perfluoroalkylpolyethers (PFPE) Fomblin Z25, Demnum S200, and Krytox 16256 were studied during sliding contact with stainless steel (440C) specimens under ultra-high-vacuum conditions. All three fluids reacted with the steel specimens during sliding. Fomblin, which has acetal linkages, decomposed under the sliding conditions and generated gaseous products, namely, COF_2 and fluorinated carbons, which were detected by a quadrupole mass spectrometer. Gaseous products were not detected for the Demnum and Krytox fluids. The amount of gaseous products from Fomblin increased with increasing sliding speed. At the end of the sliding experiments, the wear scar and the deposits on the specimens were examined by small-spot-size x-ray photoemission spectroscopy (XPS). The oxide layer on the specimen surface was removed during sliding, and metal fluorides were formed on the worn surface. The surfaces of the wear scar and the deposits were covered with adsorbed PFPE. On the basis of these results, it was concluded that the decomposition reaction of Fomblin began when the fluid contacted the fresh metal surface formed during sliding.

Introduction

Perfluoroalkylpolyethers (PFPE) have physical and chemical properties, such as low vapor pressure and high thermal and oxidative stability (refs. 1 and 2), that allow their use as vacuum pump oils (ref. 3), as lubricants for magnetic recording media (ref. 4), and for satellite applications (ref. 5). They are currently being studied for use as lubricants in advanced aircraft (ref. 6) and high-temperature gas turbine engines (ref. 7).

Interest in the use of PFPE fluids as lubricants has led to many studies (refs. 1, 2, and 6 to 12). One particular study suggested that the fresh metal surfaces formed under tribological conditions played an important role in the tribological reactions of PFPE (ref. 9). This was an interesting speculation because no experimental evidence existed to substantiate the reaction of PFPE with fresh metal surfaces. Walczak et al. (ref. 13) recently studied the chemisorption of perfluorodiethyl ether on a clean ruthenium (001) surface. The ether was found to chemisorb reversibly without decomposing.

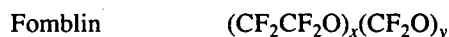
The main objective of this work was to study the effect,

under sliding conditions, of freshly formed metal surfaces on PFPE fluids used as a lubricant. Evidence was sought to substantiate any possible reaction between PFPE fluids and fresh metal surfaces.

Experiment

Materials

Three types of commercially available PFPE were used as received from the manufacturer: Fomblin Z25, Demnum S200, and Krytox 16256. The molecular formulas are as follows:



where $x/y = 0.6$ to 0.7



where $n = 50$



where $n = 66$

Fomblin is a copolymer of perfluorinated ethylene oxide and perfluorinated methylene oxide. This polymer has acetal linkages ($-\text{O}-\text{CF}_2-\text{O}-$), and the ratio of the ethylene oxide group to the methylene oxide group is 0.6 to 0.7. Demnum is a polymer of perfluorinated propylene oxide. Krytox has pendant groups ($-\text{CF}_3$) on the main chain. The properties of the fluids are summarized in table I. The molecular weights

TABLE I.—PROPERTIES OF PFPE FLUIDS

Property	PFPE fluid		
	Fomblin	Demnum	Krytox
Average molecular weight	9500	8400	11 000
Kinematic viscosity at 20 °C, cS	255	500 ± 25	2717
Viscosity index	355	210	-----
Pour point, °C	-66	-53	-15
Density at 20 °C, g/ml	1.851	1.894	1.920
Surface tension at 20 °C, dyne/cm	25	19	19
Vapor pressure, torr:			
At 20 °C	3×10^{-12}	5×10^{-11}	3×10^{-14}
At 100 °C	1×10^{-8}	1×10^{-7}	1×10^{-9}

of the fluids are similar to each other, and the vapor pressures of all three fluids are less than 10^{-10} torr (20°C).

The 440C steel specimens, whose dimensions are shown in figure 1, were polished with emery paper (#600) and diamond pastes (6, 3, and $1\text{ }\mu\text{m}$) and then rinsed with acetone in an ultrasonic bath. The surface roughness R_{max} of the flat specimen was about $0.1\text{ }\mu\text{m}$ measured by a Taylor-Hobson Talysurf 5-120 surface profilometer.

Experimental Apparatus

The experimental apparatus is shown in figure 1. A ring specimen 25.4 mm in diameter with a 12.7-mm radius of curvature was rotated against a flat specimen 19 mm in diameter through a magnetic drive assembly. In order to achieve the desired load, the rotating specimen can be moved by X and Y manipulators and then pressed to the flat specimen by the Y -axis micrometer. The load and frictional force were measured with strain gages. The equipment was mounted in a vacuum chamber that had a quadrupole mass spectrometer (UTI 100). The vacuum chamber was evacuated to less than 2×10^{-9} torr with an ion pump. While the magnetic drive assembly was being rotated, the chamber pressure increased

to 4×10^{-9} to 5×10^{-9} torr as a result of gas evolution from the assembly bearings.

Procedure

Approximately 50 mg of test fluid was placed on both metal specimens with a cotton swab. Initially, the rotor was rotated at a speed of either 3.3 or 6.6 cm/sec without specimen contact. After the pressure in the vacuum chamber reached steady state, a load was applied stepwise from 40 to 200 g in 40-g increments. The load, frictional force, and mass intensity of a certain mass number were recorded simultaneously. Typical results are shown in figure 2. As can be seen, each sliding experiment was continued for 1 min with no-load intervals of 1 min between sliding experiments. Note that the stationary flat specimen tilted with increasing load, causing the sliding position on the flat specimen to change slightly. This tilt resulted in a slight overlapping of contact regions as the load was increased.

All experiments were performed at room temperature (23°C) in an ultrahigh vacuum. Each experiment was repeated three times. The scatter for both the recorded load and the friction force measurements was within 10 percent. Coefficients of friction were calculated from the recorded load

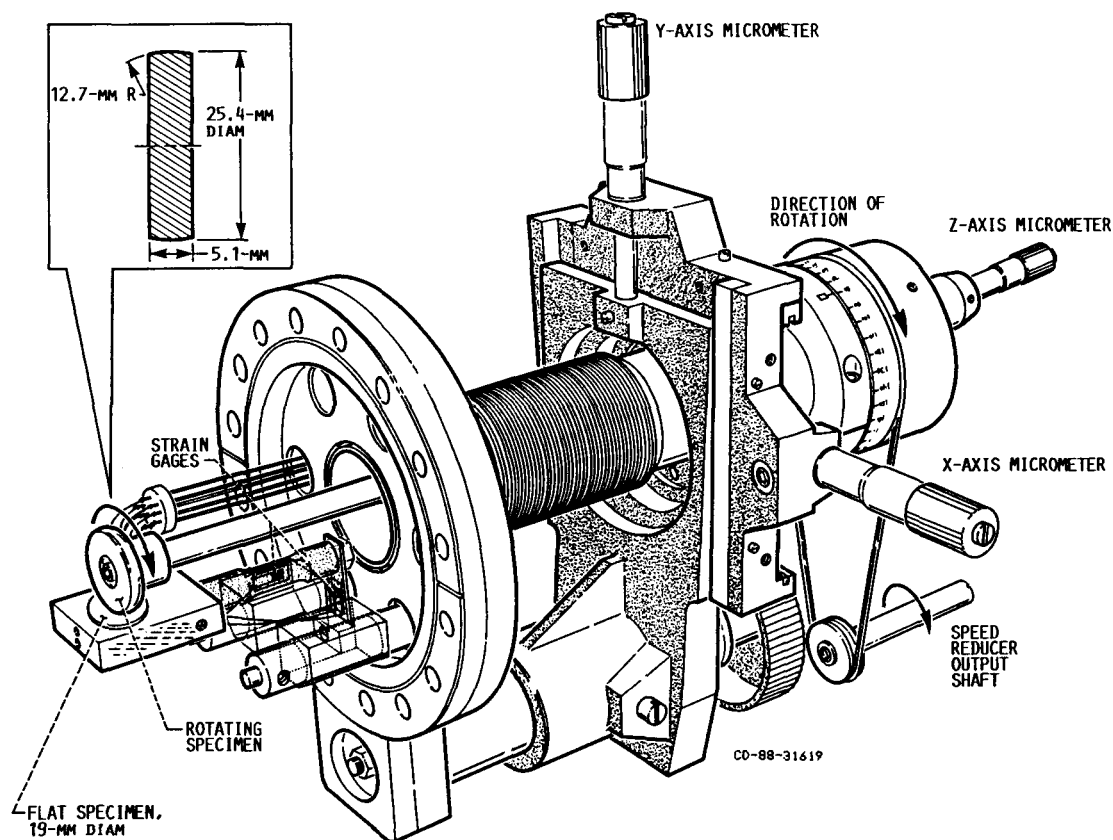


Figure 1.—Experimental apparatus.

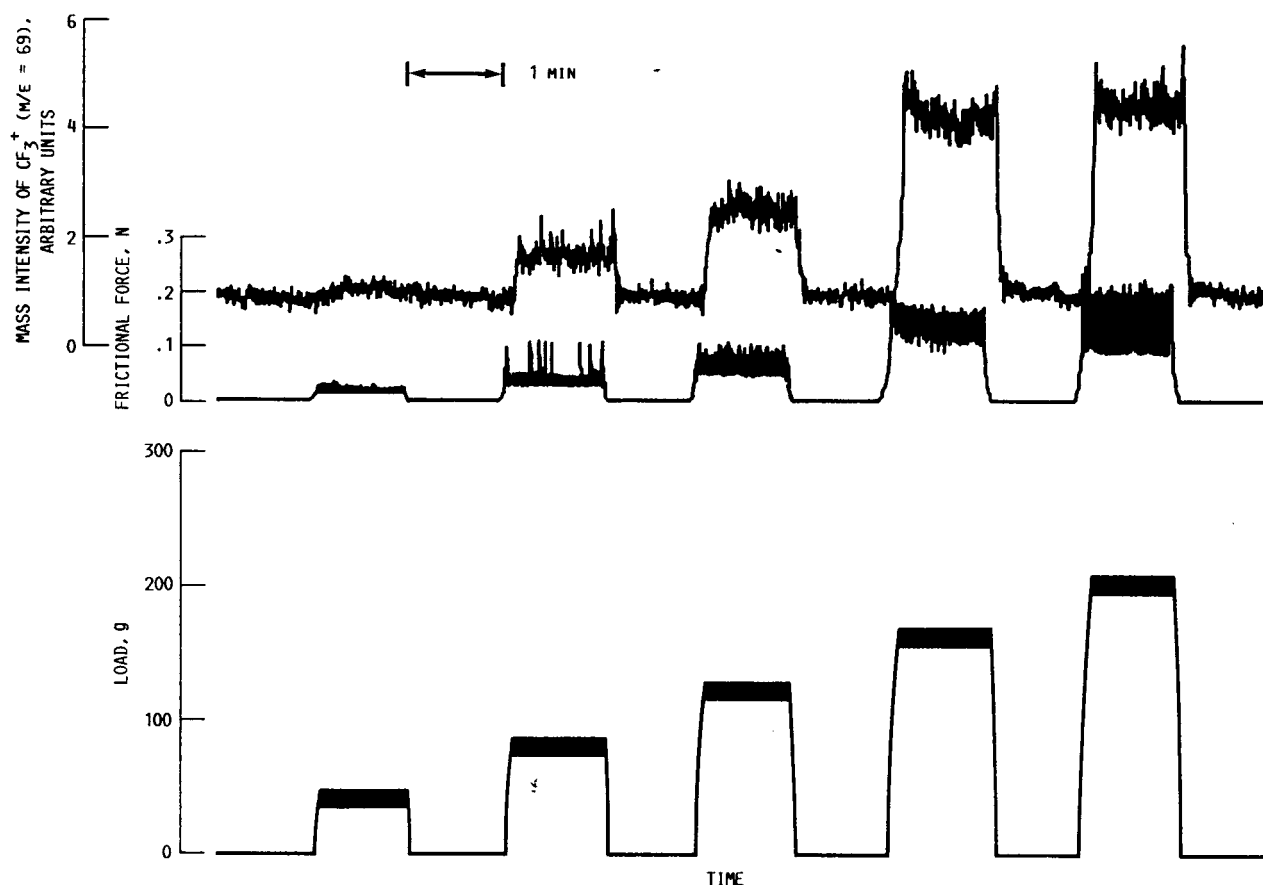


Figure 2.—Experimental procedure and typical results of frictional force and mass intensity for Fomblin on 440C steel. Sliding speed, 6.6 cm/sec

and friction force measurements. The results (shown in fig. 3) are the average of the three repeated experiments.

Chemical Analysis After Sliding Experiments

After each sliding experiment, the rotor and the flat specimens were rinsed with approximately 5 ml of 1,1,2-trichlorotrifluoroethane (TCF). The solution of PFPE (and

possible decomposition products) was analyzed by a high-pressure liquid chromatograph (HPLC) in the size-exclusion mode; the resulting chromatogram was compared with that of unreacted PFPE. Details of this HPLC analysis have been reported elsewhere (ref. 14).

The stationary specimen was examined by a small-spot-size x-ray photoemission spectroscopy (XPS) that can analyze a diameter as small as 150 μm . The x-ray source was MgK. Binding energies were calibrated with a clean copper standard ($\text{Cu}_{2p} = 932.4 \text{ eV}$, $\text{Cu}_{3p} = 74.9 \text{ eV}$). The binding energy of the C_{1s} organic contaminants was 285.1 eV. A depth profile measurement was carried out by argon ion sputtering for 2 to 4 min.

Results

Coefficients of Friction

Coefficient of friction data, obtained at a sliding speed of 6.6 cm/sec, are summarized in figure 3. The friction coefficient of Fomblin was initially about 0.05 and increased with increasing load, that of Demnum was initially about 0.1,

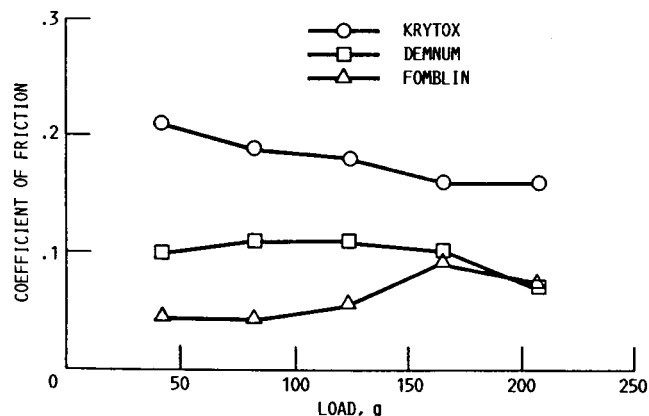


Figure 3.—Coefficient of friction for three PFPE fluids on 440C steel. Sliding speed, 6.6 cm/sec. Kinematic viscosity at 20 °C, cS: Fomblin, 255; Demnum, 500 \pm 25; Krytox, 2717.

and, for Krytox, the initial friction coefficient was high (0.2) but decreased with increasing load.

Gas Evolution During Sliding

The total pressure of the vacuum chamber and the mass spectra of evolved gaseous products were monitored under the following sliding conditions: load, 164 g; sliding speed, 6.6 cm/sec. The pressure increase during sliding with Fomblin was 1.8×10^{-9} torr. The mass spectrum of the evolved gaseous products from Fomblin is shown in figure 4. The main peak has a mass number of 47, which is a main fragment of COF_2 ($m/e = 66$). Fragments of fluorinated carbons such as CF_2 (50), CF_3 (69), C_2F_4 (100), and C_2F_5 (119) indicate that

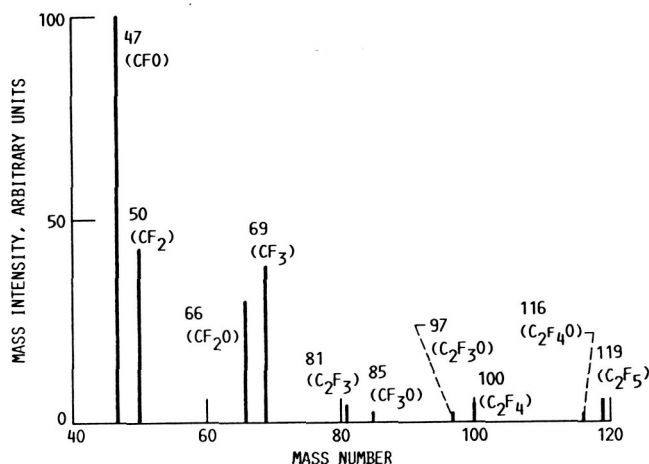


Figure 4.—Mass spectrum of evolved gas during sliding experiment with Fomblin on 440C steel. Pressure increase during sliding, 1.8×10^{-9} torr; sliding speed, 6.6 cm/sec; load, 164 g.

the evolved gaseous products contain not only a substantial quantity of COF_2 but also low-molecular-weight fluorinated carbons.

No pressure increase was observed during sliding with Demnum or Krytox. The mass spectra revealed that, during sliding, no gaseous products formed from Demnum or Krytox.

Characterization of Specimen Surface by XPS

Figure 5 shows optical micrographs of the stationary specimen surfaces at the end of a sliding experiment. During sliding, all three surfaces were scratched, with wear debris and deposits accumulating at the contact exit. XPS measurements were made at the scratched and deposit-covered areas as well as at an unworn area.

C_{1s} and O_{1s} XPS spectra for the specimen used with the Fomblin fluid are shown in figure 6. The C_{1s} spectrum (fig. 6(a)) of the unsputtered, scratched specimen surface shows that there are two types of carbon: peak B, at 285.1 eV, corresponds to organic contaminants, whereas the higher binding energy peak A, at 293.4 eV, corresponds to fluorinated carbons (ref. 4). After a 2-min ion-sputtering period, C_{1s} peak A, corresponding to fluorinated carbons, diminished in intensity, whereas C_{1s} peak B, corresponding to organic contaminants, increased in intensity. This indicates a fluorocarbon layer over a hydrocarbon (organic contaminants) layer on the scratched surface area.

The unsputtered, scratched specimen surface (fig. 6(b)) had two O_{1s} peaks. Peak B, at 530.5 eV, corresponds to a surface oxide, whereas peak A, at the higher binding energy of 535.5 eV, corresponds to a fluorinated ether (ref. 4). After sputtering, peak A diminished substantially, whereas peak B diminished only slightly. These results indicate the existence

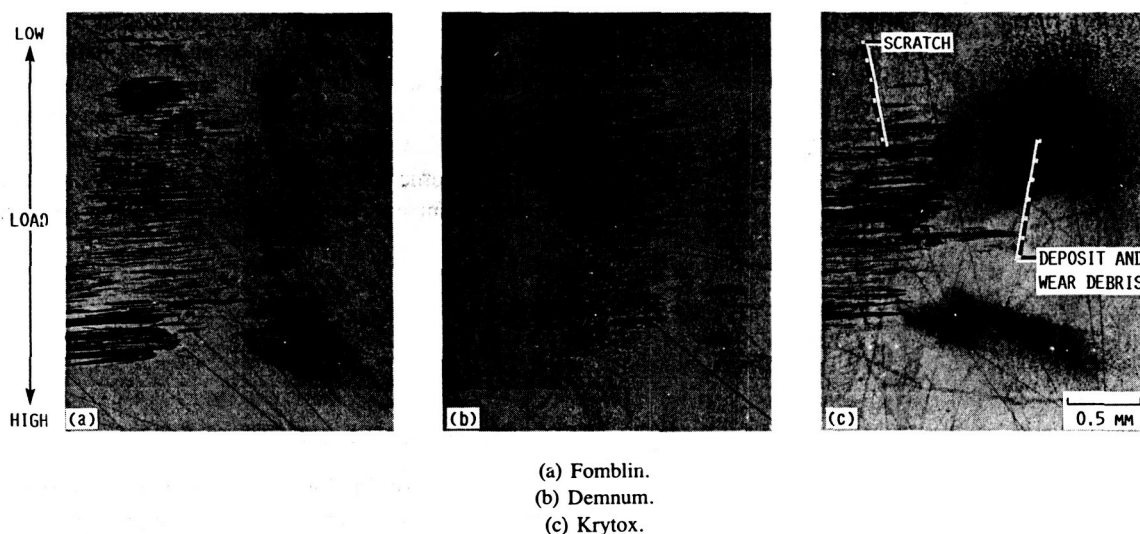


Figure 5.—Optical micrographs of specimen surfaces after sliding.

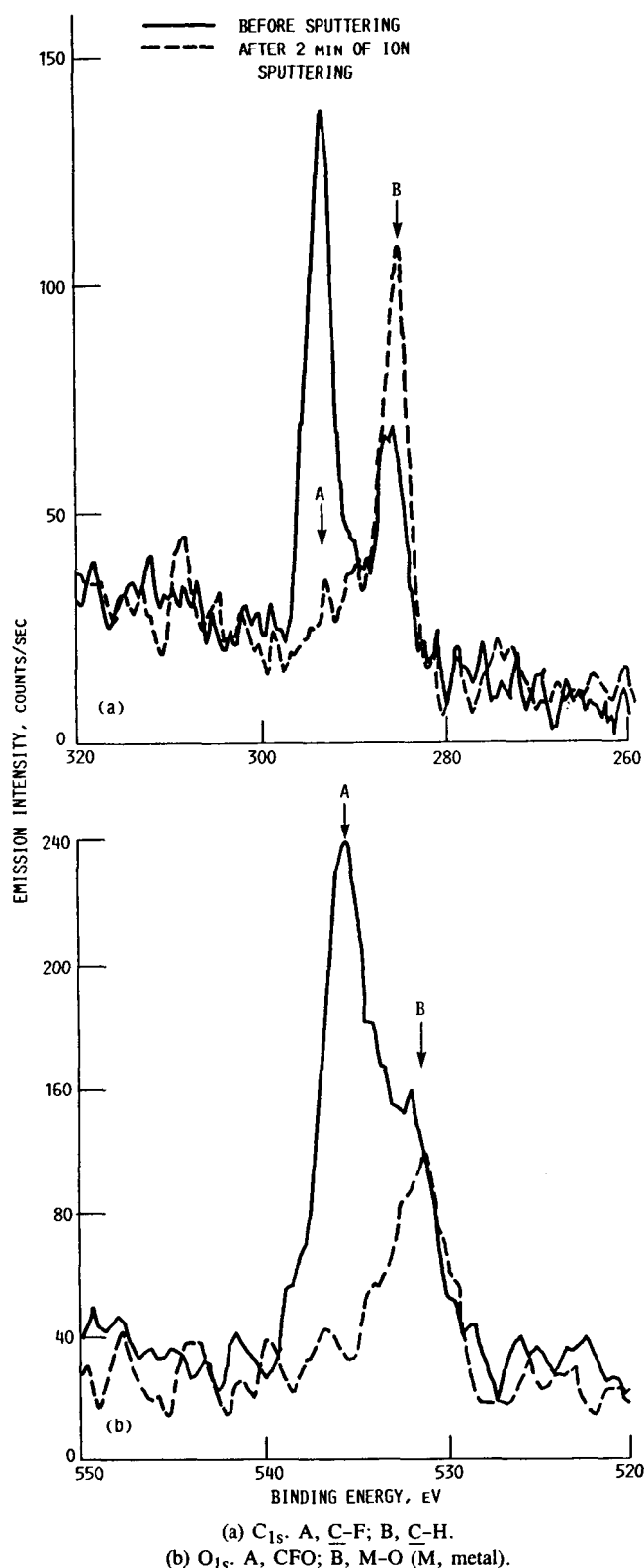


Figure 6.—XPS spectra of scratched area of 440C steel specimen after sliding with Fomblin.

of a fluorinated ether layer over the surface oxide of the scratched area. In this case, the fluorinated ether is an adsorbed fluorinated carbon substance that may be unreacted Fomblin or a fluorinated product of Fomblin decomposition.

F_{1s} XPS spectra for the specimens used with all three fluids are shown in figure 7. Peak A, at 689.1 eV, and peak B, at 685.6 eV, are measured. F_{1s} peak A corresponds to fluorinated carbons (ref. 4), whereas peak B may be assigned to metal fluorides (ref. 15). In all three cases, F_{1s} peak A diminished after sputtering. F_{1s} peak B increased in intensity, after sputtering, for the Fomblin and Demnum fluids (figs. 7(a) and (b)); for the Krytox fluid, the peak intensity was initially high (fig. 7(c)) and a small decrease in peak intensity occurred after 4 min of sputtering. These results show (1) that metal fluoride is formed at the sliding contacts for all three fluids and (2) that the adsorbed fluorocarbon layer on the Krytox-lubricated surface is thinner than the layers formed from Fomblin or Demnum, as judged by the changes of F_{1s} peaks with sputtering time.

Table II summarizes the intensity changes of the O_{1s} metal oxide peak during the sliding experiments with all three fluids for scratched and unworn specimen surface areas. The intensity of the O_{1s} peak at the unworn area did not decrease with sputtering time, whereas the peak intensity at the scratched area was small and did decrease with sputtering time. These results indicate that the oxide layer on the metal specimens was removed during sliding.

The wear debris from all three fluids contained both perfluorinated polyether and metal fluorides.

High-Pressure Liquid Chromatograph Analysis

HPLC analysis of the PFPE fluids extracted with TCF solvent from the specimen surfaces revealed no decomposition products or changes in the molecular weight distribution of the original PFPE fluids. This resulted from the fact that either the amount of decomposition products in the liquid phase was too small to detect by HPLC or that the products were insoluble

TABLE II.—INTENSITY OF O_{1s} METAL OXIDE PEAK

Sputtering time, min	PFPE fluid					
	Fomblin		Demnum		Krytox	
	S ^a	U ^b	S ^a	U ^b	S ^a	U ^b
	Intensity, counts/sec					
0	120	490	350	530	440	680
2	100	720	400	680	550	810
4	120	670	370	700	520	730

^aS, scratched area.

^bU, unworn area.

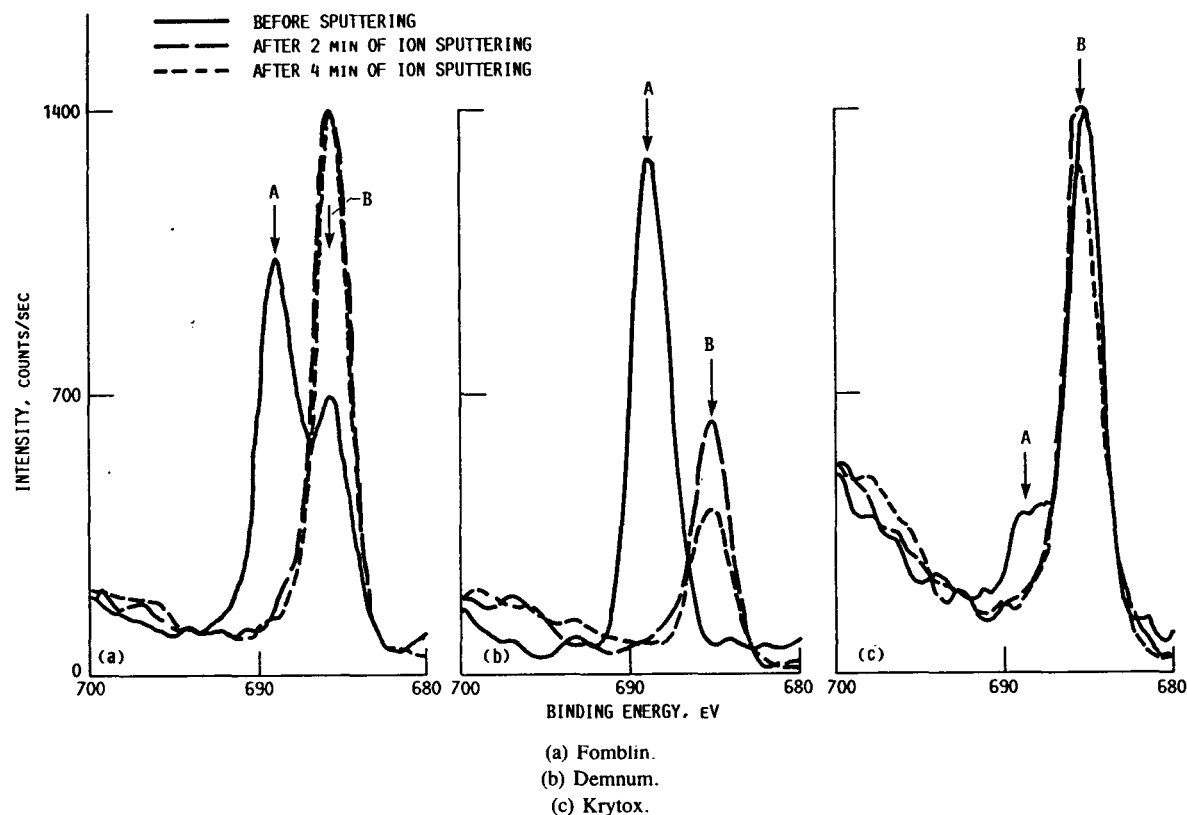


Figure 7.— F_{1s} XPS spectra of scratched area after sliding with Fomblin, Demnum, and Krytox. A, C-F; B, M-F (M, metal).

Additional Experimental Results With Fomblin

A series of experiments was conducted to determine the way in which different sliding conditions affected the decomposition of Fomblin. A mass spectrometer was used to monitor the mass intensity change of mass numbers 47 (COF^+), 66 (COF_2), and 69 (CF_3). Figure 8 shows the mass intensity of COF^+ as a function of load and sliding speed. The higher the sliding speed and load, the greater the amount of evolved gas. Similar results were obtained for the gaseous products having mass numbers 66 and 69. In addition to the mass intensity measurements, frictional force was measured as a function of load and sliding speed. The results are presented in figure 9.

Figure 10 shows a typical mass spectrum monitoring COF_2 ($m/e = 66$) during a sliding experiment with Fomblin. The interesting feature in this spectrum is the slow decrease in the COF_2 peak intensity (the tailing behavior) after the sliding experiment was terminated. This indicates either the possible accumulation of COF_2 in Fomblin or the continuing decomposition of Fomblin after sliding terminated.

Fomblin film thickness between the two 440C steel specimens was calculated as a function of load and sliding speed (ref. 16). The results from figure 11 show that the Fomblin film thickness decreases to less than $0.2 \mu m$ as the load increases for both sliding speeds. Since the R_{max} of the

flat 440C steel specimen was about $0.1 \mu m$, these results show that boundary lubricating conditions (mechanical contact of the specimens occurs at the sliding contact area) exist at the higher loads, where gaseous product formation is greatest.

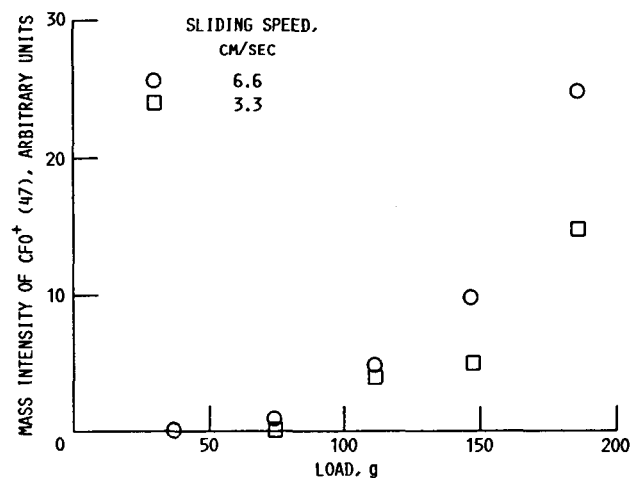


Figure 8.—Effects of load and sliding speed on gas evolution from Fomblin.

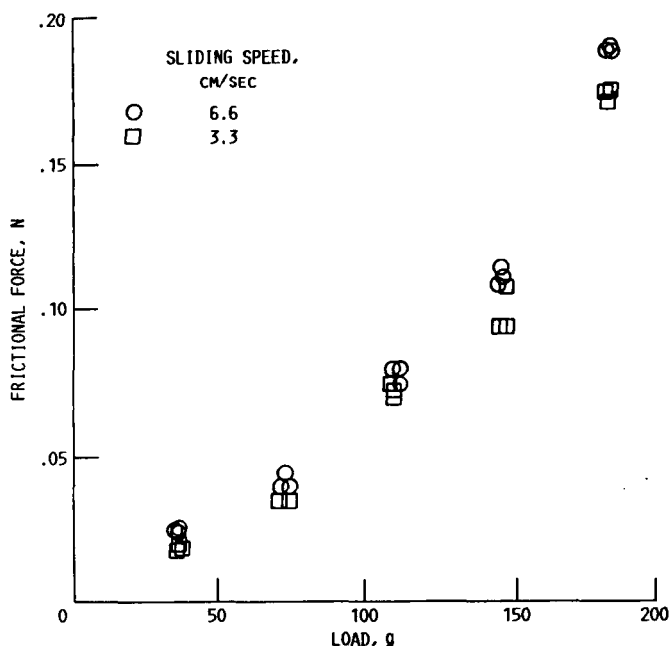


Figure 9.—Effects of load and sliding speed on frictional force for 440C steel lubricated with Fomblin.

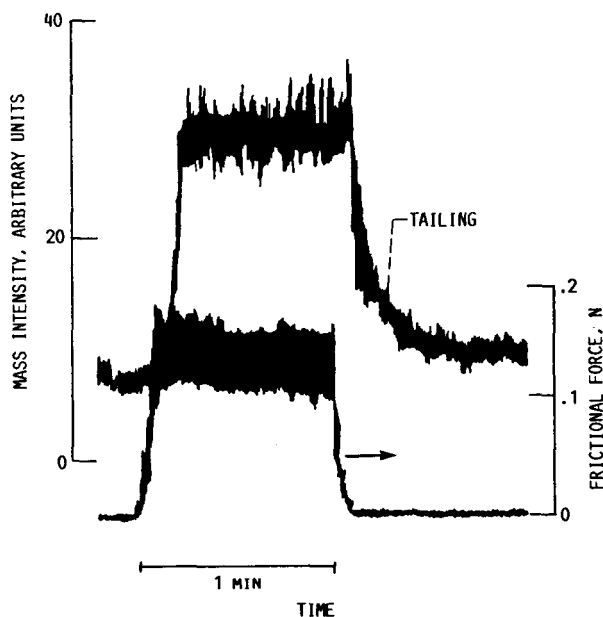


Figure 10.—Tailing behavior of gas evolution of COF_2 ($m/e = 66$) from Fomblin. Load, 164 g; sliding speed, 6.6 cm/sec.

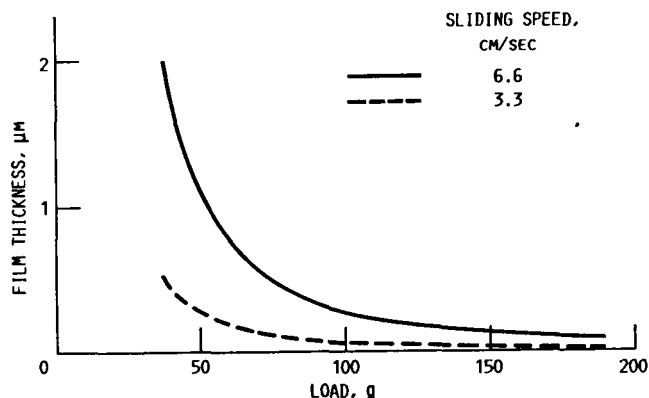


Figure 11.—Film thickness of Fomblin under sliding conditions (ref. 17).

Discussion

The experimental results showed that Fomblin reacted at the sliding contacts, thereby producing large quantities of COF_2 and smaller quantities of various low-molecular-weight fluorocarbons. XPS measurements gave evidence that Demnum and Krytox also reacted at the sliding contacts. Before all the experimental results are discussed in detail, the main causes of tribochemical reactions must be clarified.

It is generally accepted that the two main causes of tribochemical reactions are (1) heat generation at the sliding contacts and (2) high chemical activity on fresh metal surfaces generated by mechanical shear. Are the tribochemical reactions of the three PFPE fluids the result of one, or the other, or both? The question is asked in light of several studies conducted on the thermal and thermal-oxidative decomposition of PFPE fluids. It was reported in one study (ref. 12) that Fomblin decomposed at 288°C in the presence of metals under an oxygen atmosphere. In another study it was reported that Fomblin was catalytically degraded by certain metal oxides at 345°C under both air and nitrogen atmospheres (ref. 14). Krytox was used on 440C steel specimens, under boundary lubricating conditions, in a wear test apparatus (ref. 9). The author concluded that Krytox decomposition occurred as the result of high sliding contact temperatures.

In these studies, the sliding conditions are mild (i.e. low loads and low sliding speeds). Therefore, the frictional heat generation can be assumed to be proportional to the measured frictional force and sliding speed (ref. 17). From figure 9, the frictional force at the 74-g load, with a sliding speed of 6.6 cm/sec, is approximately 0.039 N. The frictional force at the 113-g load, with a sliding speed of 3.3 cm/sec, is 0.074 N. The calculated heat generation at the 74-g load is therefore 2.6×10^{-3} J/sec, whereas at the 113-g load, it is 2.4×10^{-3} J/sec. Because the heat generation for these two conditions is approximately the same, the amount of gaseous products from Fomblin decomposition would be expected to be about the same also. However, the gaseous products at the higher load are about five times those at the lower load. Under

these sliding conditions, frictional heat generation does not significantly affect PFPE decomposition at the sliding contacts.

It can be concluded, then, that all three PFPE fluids reacted with the fresh metal surfaces formed during the sliding experiments. In all three cases, XPS measurements showed an adsorbed fluorocarbon layer over a metal fluoride layer at the scratched sliding contact area. Wear debris, consisting of fluorinated polyethers and metal fluorides, accumulated at the sliding contact exit for all three PFPE fluids.

It appears from the results summarized in table II that, during sliding, surface metal oxide is worn away, thus generating a fresh metal surface that reacts with the PFPE fluids. The products of these reactions appear to be a metal fluoride (formed over the fresh metal surface) and a fluorocarbon (insoluble in TCF solvent) adsorbed over the metal fluoride. For Fomblin, gaseous products (primarily COF_2) are formed. As sliding continues, the metal fluoride and the adsorbed fluorocarbon are worn away, accumulating in the form of wear debris at the sliding contact exit.

At the end of a sliding experiment with Fomblin, it was noticed that mass detection of COF_2 continued for some time (fig. 10). If this were due to COF_2 accumulation in the Fomblin fluid, then the mass detection of COF_2 at the beginning of the sliding test should have been a slow increase from zero mass intensity to final, steady-state mass intensity. This was not the case. The mass intensity of COF_2 increased very rapidly. In addition, Fomblin decomposition appeared to continue after sliding terminated. Because any fresh metal surface formed during sliding should immediately react with Fomblin to produce metal fluoride, the tailing behavior (fig. 10) may have been due to catalytic activity of the metal fluoride. Iron fluoride (FeF_3) is reported as a Lewis acid that acts as a catalyst on the decomposition of Krytox (ref. 9) and Fomblin (ref. 14).

The differences in reactivity between the three PFPE fluids are due to their molecular structures. Fomblin has acetal linkages ($-\text{O}-\text{CF}_2-\text{O}-$) in its structure. Since gaseous decomposition products (i.e., COF_2) were detected only for Fomblin, it can be concluded that the acetal linkages are the primary cause of gas product formation. Krytox has pendant groups ($-\text{CF}_3$) in its molecular structure. It is interesting to note that the thinnest fluorocarbon layer was formed from Krytox, whereas the thickest layer was formed from Demnum, which has a structure lacking both the acetal and pendant groups.

Conclusions

Sliding experiments with three types of commercially available perfluoroalkylpolyethers (PFPE) were carried out under ultra-high-vacuum conditions at room temperature. From the results, the following conclusions can be drawn:

1. All three PFPE fluids reacted with 440C stainless steel

under sliding conditions, generating metal fluorides and a fluorocarbon product at the scratched sliding contact area. Gaseous products, COF_2 and fluorinated carbons, were formed only from Fomblin, which has acetal linkages ($-\text{O}-\text{CF}_2-\text{O}-$).

2. A fresh metal surface was generated from the mechanical shear of the original surface oxide layer. The fresh metal surface initiated the decomposition of the PFPE fluids.

3. The metal fluoride and fluorocarbon layers were worn away, accumulating in the form of wear debris at the sliding contact exit.

4. The metal fluorides showed catalytic activity for the formation of COF_2 from Fomblin.

Lewis Research Center
National Aeronautics and Space Administration
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